

Results of the 16th Proficiency Test on Pesticide Residues in olive oil



Tiziana Generali¹, Patrizia Stefanelli¹, Silvana Girolimetti¹, Danilo Attard Barbini¹

¹ Dipartimento di Ambiente e Salute, Istituto Superiore di Sanità, Rome, Italy



1 blank oil + 1 spiked oil
Homogeneity and stability tests performed
ISO 13528:2015

List of pesticides Reporting Limit (RL) 0.05 mg/kg	
Chlorpyrifos	Metidathion
Chlorpyrifos-methyl	Omethoate
lambda-Cyhalothrin	Oxyfluorfen
Deltamethrin	Phosalone
Diazinon	Procymidone
Diflufenican	Quinalfos
Dimethoate	Kresoxim methyl
alfa-Endosulfan	Simazine
beta-Endosulfan	Terbutylazine
Endosulfan sulphate	Tolclofos methyl
Fenitrothion	Trifloxystrobin
Fenoxy carb	Trifluralin
Fenthion	
Fenthion sulfone	
Fenthion sulfoxide	

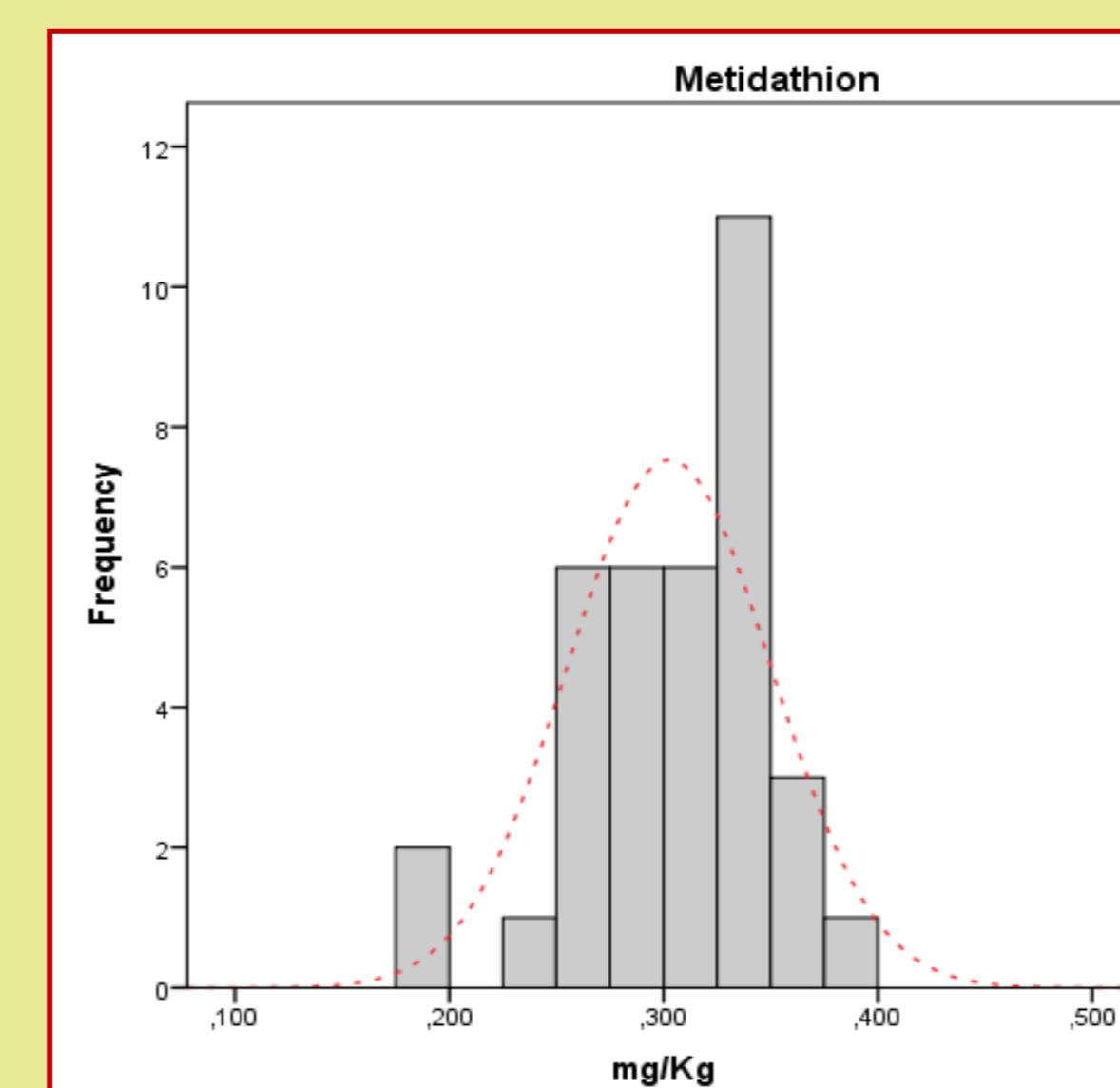
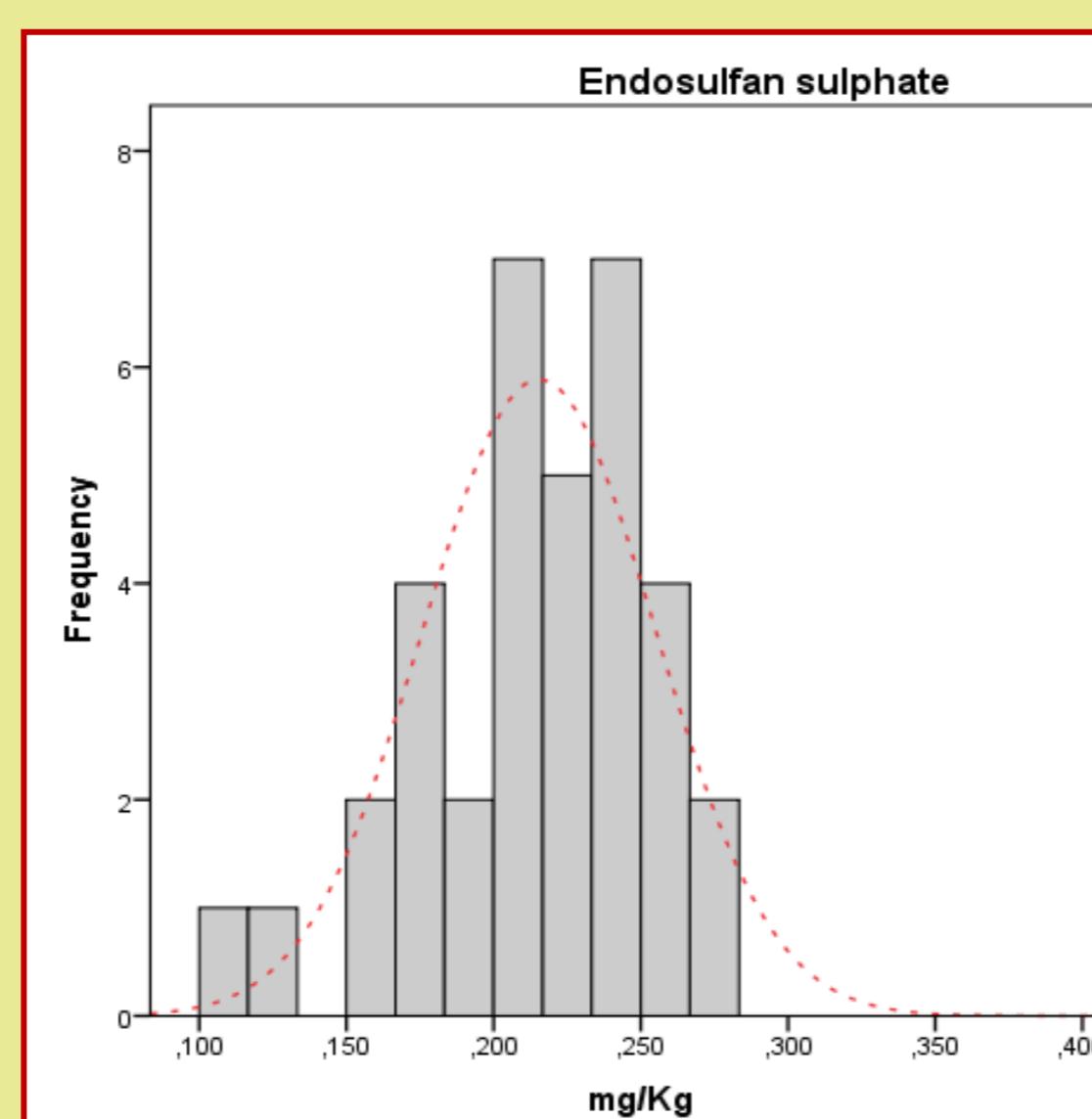
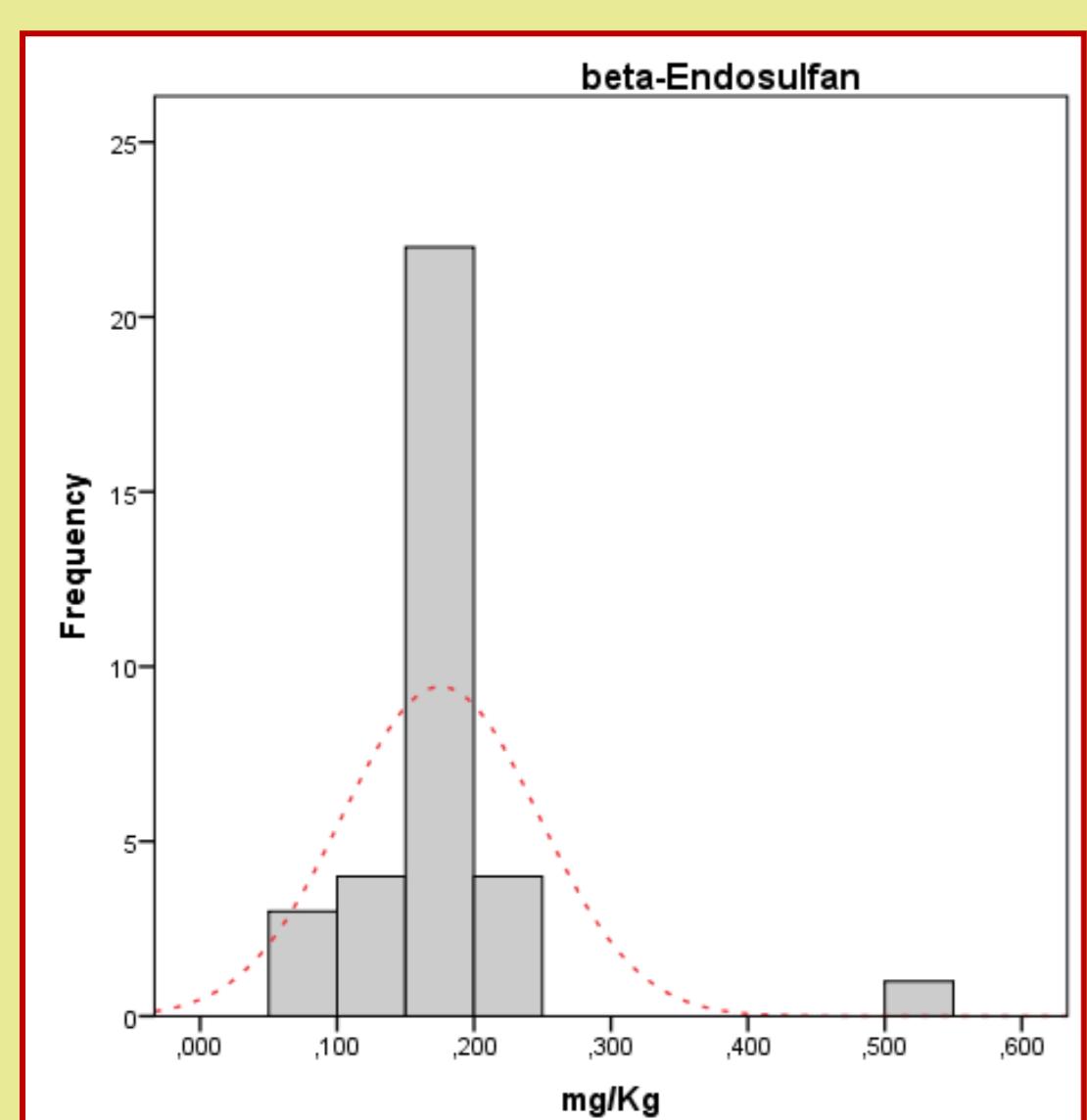
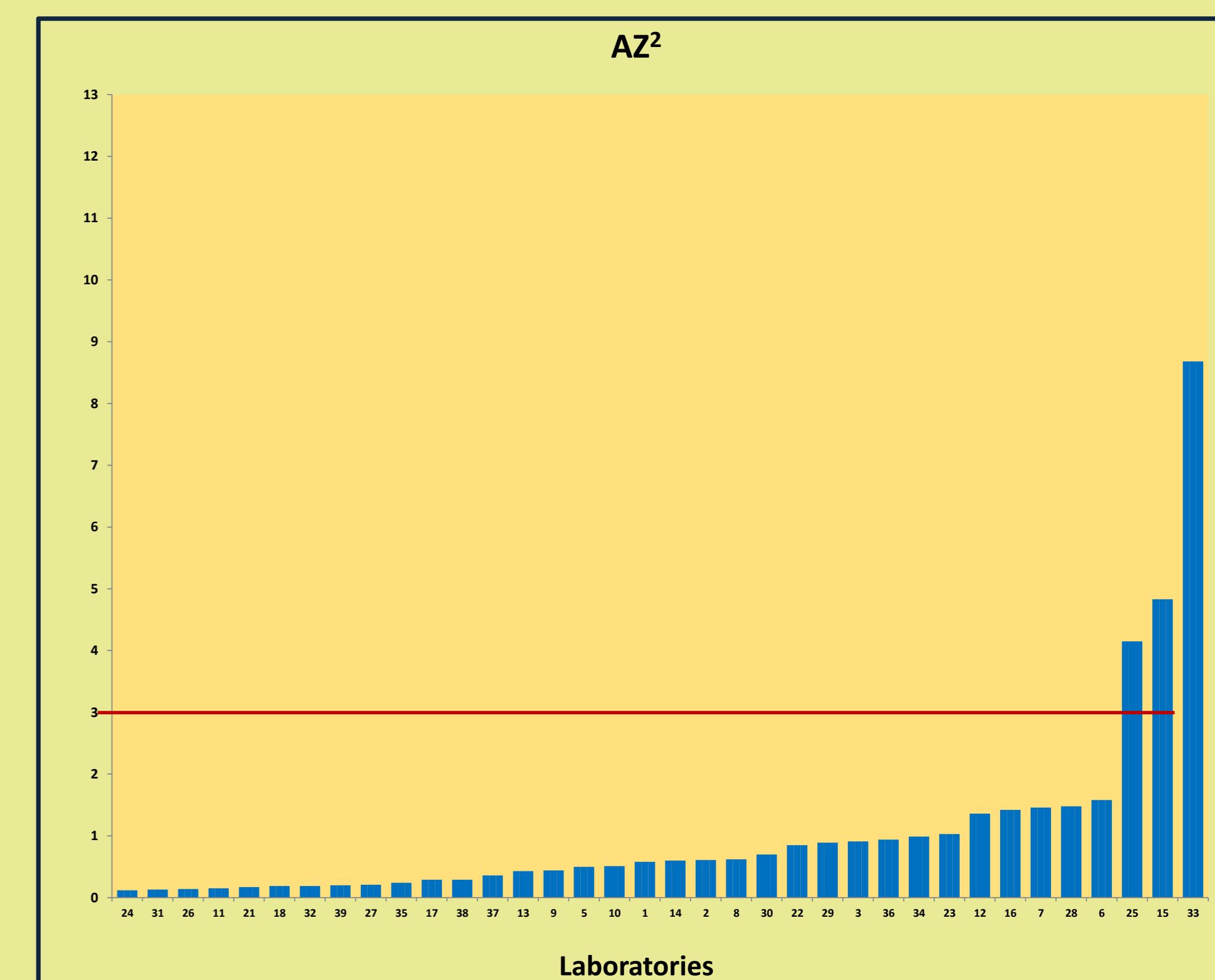
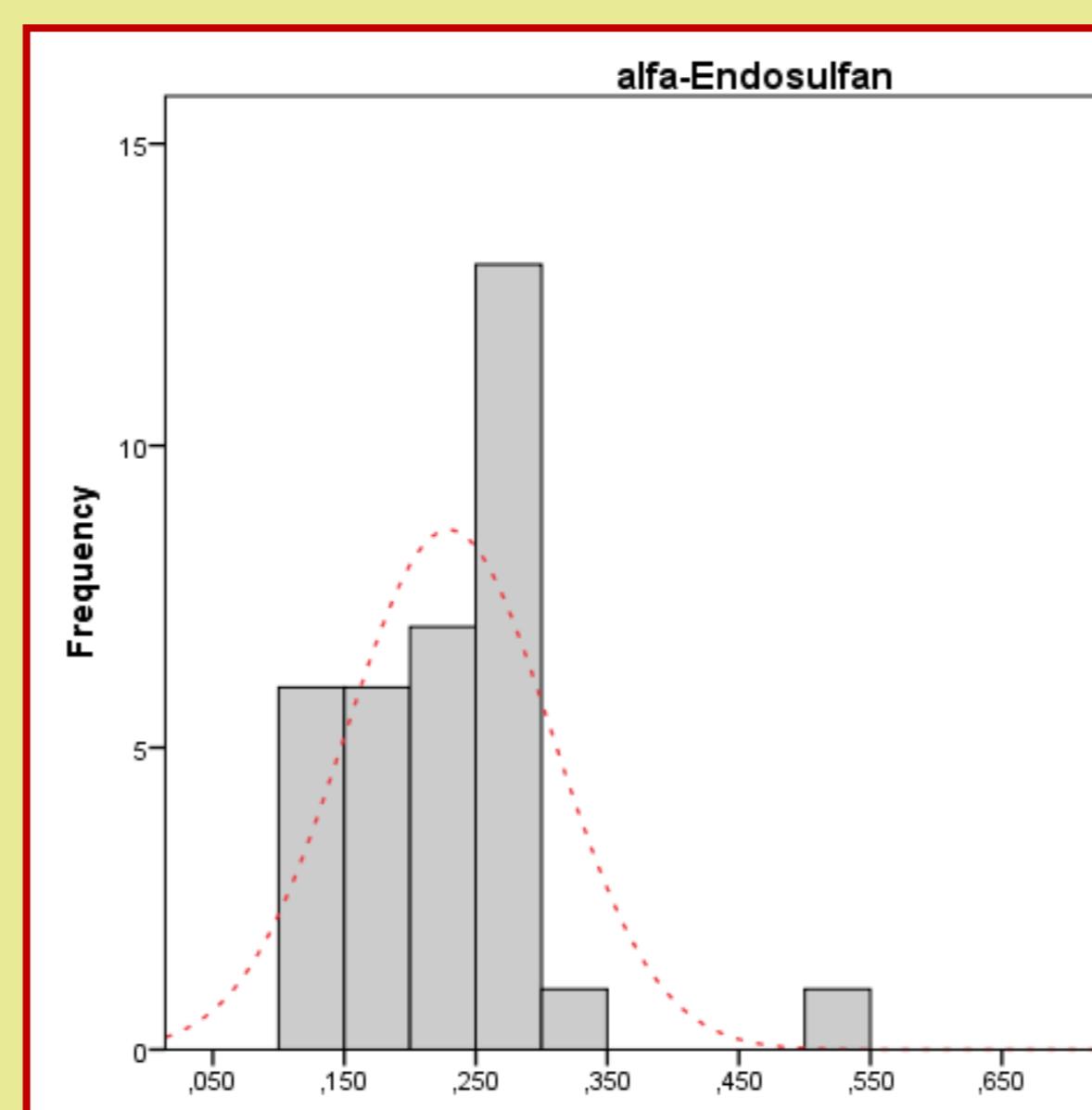
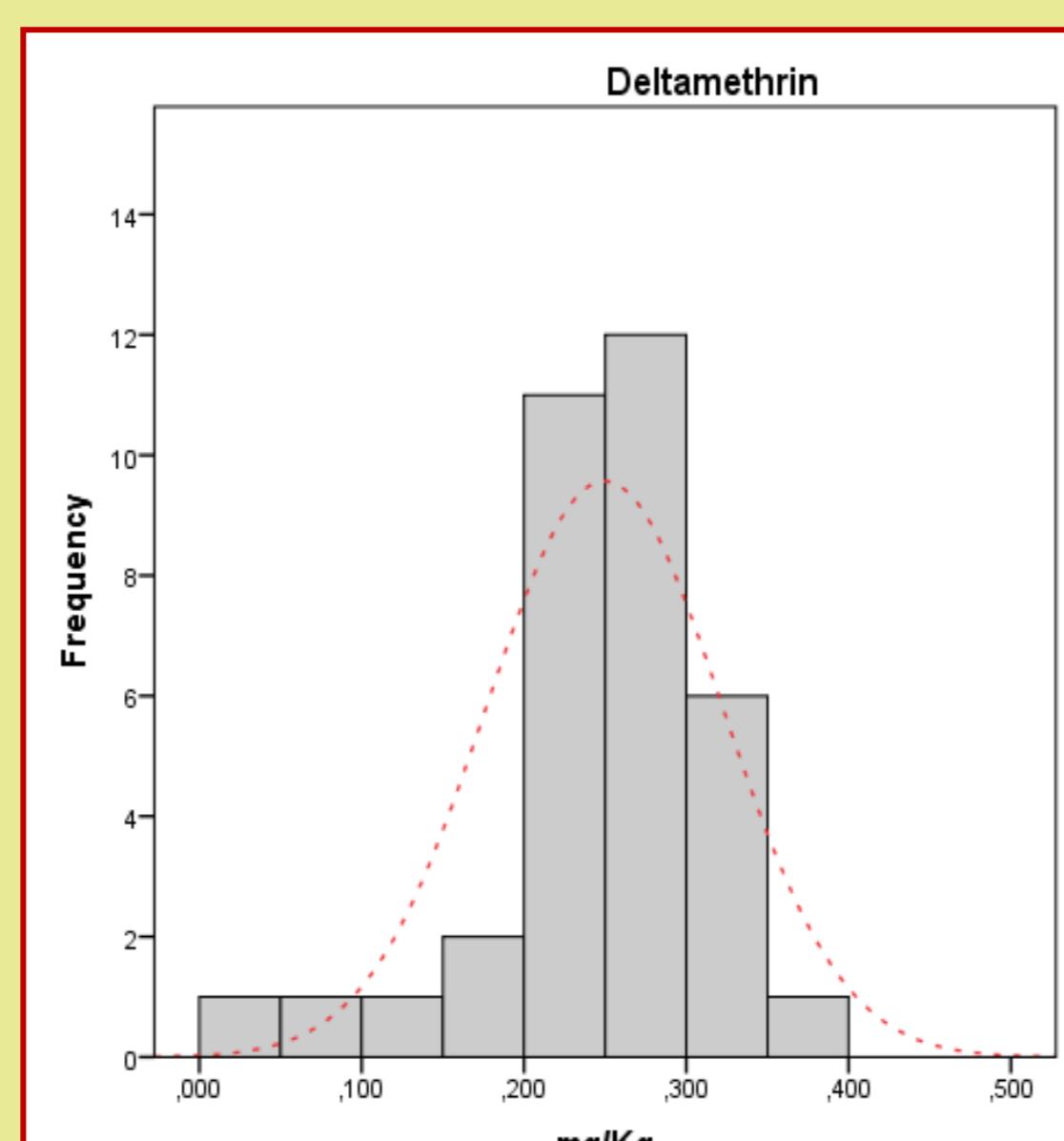
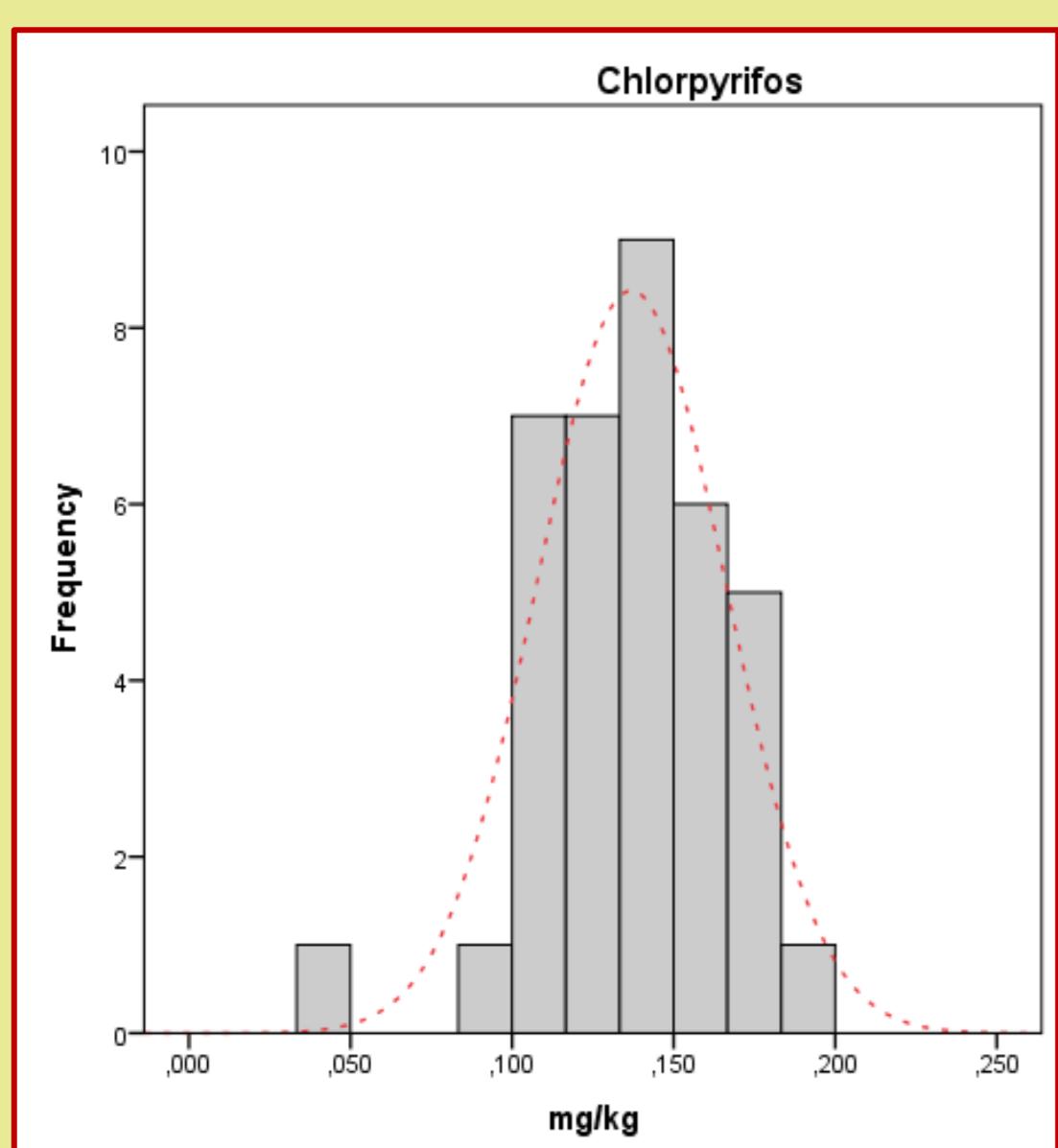
List of z-scores

Lab	Chlorpyrifos	Deltamethrin	Alpha Endosulfan	Beta Endosulfan	Endosulfan sulphate	Metidathion
1	-1.1	0.5	-0.5	0.4	1.2	-0.4
2	0.7	1.1	1.0	0.6	0.7	0.3
3	-1.0	-0.3	-1.5	-1.0	0.7	0.8
5	-0.3	-1.1	-1.2	-0.2	-0.2	-0.4
6	-0.9	-2.4	0.5	-0.3	0.0	-1.6
7	-0.8	0.5	-1.9	-1.8	-0.8	-0.6
8	1.1	0.9	1.1	0.0	0.5	0.5
9	0.7	0.8	0.8	0.5	-0.8	0.0
10	-0.6	-0.7	-1.1	-0.6	0.6	0.5
11	0.1	-0.8	0.4	0.1	0.2	-0.1
12	-0.7	0.5	-2.0	-1.6	-0.7	-0.6
13	1.0	0.7	0.6	0.4	0.3	0.7
14	0.4	1.4	0.1	0.0	0.5	1.1
15	-2.7	-2.5			-1.7	-1.7
16	0.0	1.9	0.8	0.7	-1.9	0.4
17	0.6	0.3	0.4	0.8	0.7	-0.1
18	-0.3	-0.3	-0.8	0.3	0.2	0.4
21	0.3	0.2	0.6	0.7	-0.1	-0.1
22	1.5	0.4	1.2	1.0	-0.1	0.5
23	0.8	1.4	1.1	1.4	0.4	0.5
24	-0.1	-0.3	0.3	0.2	-0.5	-0.7
25	1.2	-3.8	1.6	1.5		0.2
26	0.0	0.3	0.6	0.2	0.5	0.3
27	0.6	-0.3	0.8	0.2	-0.2	-0.3
28	-0.5	0.4	-2.0	-1.8	-1.1	0.1
29	-1.3	-0.9			-1.0	0.2
30	-0.3	-1.0	-1.7	-0.1	-0.2	0.4
31	0.1	0.0	-0.7	-0.1	0.5	0.1
32	0.1	-0.5	-0.8	-0.4	0.0	0.3
33	1.0	0.0	5*	5*	1.0	-0.3
34	-0.8	-0.7	-1.7	-1.2	-0.3	-0.6
35	1.0	-0.1	0.4	0.0	0.1	0.5
36	-0.3	-0.3	-0.1	-2.2	-0.3	-0.7
37	0.3	0.5	0.6	-0.2	-0.9	-0.7
38	0.2	0.5	0.8	-0.3	0.6	0.6
39	-0.2	0.4	0.2	-0.3	0.6	-0.7

$$Z - \text{score} = \frac{x - X(\text{robust mean})}{\sigma_{\text{EPUT}}}$$

$$|AZ^2| = \frac{\sum_{i=1}^n |Z_i| |Z_i|}{n}$$

Three compounds Deltamethrin, alpha-Endosulfan and beta-Endosulfan presented distributions asymmetric



The compared results of two compounds analyzed in the 2015 and 2016 PTs showed an improvement in the performance of second PT

Methods

25 laboratories used the QuEChERS methodology or methods based on QuEChERS

6 laboratories used in house methods with an extraction step followed by a clean-up phase using the GPC technique, alumina cartridge or using combination of different materials as extrelut + silica+C₁₈ as SPE or PSA+GCB+C₁₈

2 laboratories followed the method EURL-FV (2012-M6). "Validation Data of 127 Pesticides Using a Multiresidue Method by LC-MS/MS and GC-MS/MS in Olive Oil"

1 laboratory Lentza Rizos, Journal of Chromatography A 921 (2001) 297-304 1 laboratory UNI EN 1528

